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Indian Standard

SPECIFICATION FOR AMMONIUM BROMIDE, PURE AND ANALYTICAL REAGENT

(First Revision)

UDC 661·528·341 : 54-4

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 4 *November* 1985

Indian Standard

SPECIFICATION FOR AMMONIUM BROMIDE, PURE AND ANALYTICAL REAGENT

(First Revision)

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Indian Standard

SPECIFICATION FOR AMMONIUM BROMIDE, PURE AND ANALYTICAL REAGENT

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 18 March 1985, after the draft finalized by the Acids, Alkalis and Halides Sectional Committee had been approved by the Chemical Division Council.

0.2 Ammonium bromide is used in the preparation of silver bromide in the chemical industry, in medicine and as an analytical reagent.

0.3 This standard was first published in 1964. It has been revised in the light of experiences gained over all these years regarding methods of analysis. In this revision, one more method for the determination of iron which is more sensitive and requires less reagents have been incorporated as a referee method. Method for the determination of heavy metals has been modified, and a new requirement of arsenic for analytical reagent grade has also been specified.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS:2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for ammonium bromide, pure and analytical reagent.

2. GRADES

2.1 The material shall be of two grades, namely, pure and analytical reagent.

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*Rules for rounding off numerical values (*revised*).

3. REQUIREMENTS

3.1 Description — The material shall be in the form of a small colourless crystals or a white crystalline powder, free from foreign matter and visible impurities. It is slightly hygroscopic and slowly becomes yellowish in air and should be kept in well-closed containers.

3.2 The material shall comply with the requirements given in Table 1 when tested according to the methods specified in Appendix A. Reference to the relevant clauses of Appendix A is given in col 5 of the table.

TABLE 1 REQUIREMENTS FOR AMMONIUM BROMIDE, PURE AND ANALYTICAL REAGENT

SL No.	CHARACTERISTIC	REQUIREMENT FOR		METHOD OF TEST (REF TO CL No. IN APPENDIX A)
		Pure Grade	Analytical Reagent Grade	
(1)	(2)	(3)	(4)	(5)
i)	Ammonium bromide, percent by mass of dried material, *Min*	98·0	99·5	A-2
ii)	Matter insoluble in water, percent by mass, *Max*	—	0·005	A-3
iii)	*p*H of aqueous solution, *Min*	—	4·8	A-4
iv)	Chlorides (as Cl), percent by mass, *Max*	0·6	0·2	A-5
v)	Bromates (as BrO_3) percent by mass, *Max*	0·001	0·001	A-6
vi)	Iodides	—	To pass test	A-7
vii)	Heavy metals (as Pb), percent by mass, *Max*	0·001	0·000 5	A-8
viii)	Iron (as Fe), percent by mass, *Max*	0·008	0·000 2	A-9
ix)	Sulphates (as SO_4) percent by mass, *Max*	0·06	0·01	A-10
x)	Moisture, percent by mass, *Max*	1·0	0·5	A-11
xi)	Sulphated ash, percent by mass, *Max*	0·1	0·01	A-12
xii)	Arsenic (as As_2O_3), parts per million, *Max*	10	1	A-13
xiii)	Tarry matter	To pass Test	—	A-14
xiv)	Barium	To pass Test	—	A-15

4. PACKING AND MARKING

4.1 Packing — Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in clean galvanized iron drums lined with polyethylene, or in glass bottles.

4.2 Marking — The containers shall be marked with the name and grade of the material; name of the manufacturer and recognized trade-mark, if any, mass of the material in the container; and batch number and the date of manufacture. The containers of the analytical reagent grade material shall also be labelled with the analytical data for the characteristics given in col 4 of Table 1.

4.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 Preparation of Test Samples — Representative test samples of the material shall be prepared as prescribed in Appendix B.

5.2 Number of Tests

5.2.1 Test for the determination of ammonium bromide shall be performed on each of the individual samples (*see* **B-3.3**).

5.2.2 Tests for the determination of all other characteristics given in Table 1 shall be performed on the composite sample (*see* **B-3.2**).

5.3 Criteria for Conformity

5.3.1 *For Individual Samples* — From the test results for ammonium bromide, the mean ($\bar{X}$) and range (R) of test results shall be computed (range being defined as the difference between the maximum and minimum values of test results).

5.3.1.1 The lot shall be declared as conforming to the requirements for ammonium bromide if the value of ($\bar{X} - 0{\cdot}6\ R$) as calculated from the test results is equal to or greater than the corresponding value given for the relevant grade in Table 1.

5.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant requirement specified.

APPENDIX A

(*Clause* 3.2)

METHODS OF TEST FOR AMMONIUM BROMIDE, PURE AND ANALYTICAL REAGENT

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS: 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF AMMONIUM BROMIDE

A-2.1 Reagents

A-2.1.1 *Standard Silver Nitrate Solution* — 0·1 N.

A-2.1.2 *Concentrated Nitric Acid* — *See* IS: 264-1976†.

A-2.1.3 *Nitrobenzene*

A-2.1.4 *Ferric Alum Indicator Solution* — Saturated.

A-2.1.5 *Standard Ammonium Thiocyanate Solution* — 0·1 N.

A-2.2 Procedure — Dry the material to constant mass at 105 ± 2°C. Weigh accurately about 0·4 g of the dried material and dissolve in 50 ml of water. Add with a pipette 50 ml of standard silver nitrate solution, 2 ml of concentrated nitric acid, 15 ml of nitrobenzene and finally 2 ml of ferric alum indicator solution. Titrate the solution with standard ammonium thiocyanate solution to the first persistent colour change.

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*Specification for water for general laboratory use (*second revision*).

†Specification for nitric acid (*second revision*).

A-2.3 Calculation

$$\text{Ammonium bromide (as } NH_4Br \text{), percent by mass of the dried material} = \frac{0{\cdot}979\,6\,(50-V)}{M}$$

where

V = volume in ml of standard ammonium thiocyanate solution used in the titration, and

M = mass in g of the dried material taken for the test.

NOTE — Presence of chlorides increases the results for ammonium bromide and the results shall therefore be corrected. One gram of chloride (as Cl) is equivalent to 2·76 g of ammonium bromide.

A-3. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-3.1 Procedure — Accurately weigh about 20 g of the material and dissolve it in about 150 ml of water in a beaker. Cover the beaker and digest for one hour on a water-bath. Filter the solution through a tared filter paper or tared Gooch or sintered glass crucible (G. No. 4) and wash the residue thoroughly with water. Dry the filter paper or the crucible along with residue to constant mass at 105 to 110°C.

A-3.2 Calculation

$$\text{Matter insoluble in water, percent by mass} = 100\,\frac{M_1}{M_2}$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the material taken for the test.

A-4. DETERMINATION OF *p*H OF AQUEOUS SOLUTION

A-4.1 Procedure — Dissolve 5·0 g of the material in 100 ml of freshly boiled and cooled water (the water shall be protected with a soda lime tube while cooling). Determine the *p*H of the solution using a suitable *p*H meter (*see* IS : 5741-1970*).

A-5. TEST FOR CHLORIDES

A-5.1 Apparatus

A-5.1.1 *Nessler Cylinders* — 50 ml capacity.

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*Method for determination of *p*H.

A-5.2 Reagents

A-5.2.1 *Dilute Nitric Acid* — 1 : 2.

A-5.2.2 *Hydrogen Peroxide* — Dilute one volume of 30 percent hydrogen peroxide with one volume of water.

A-5.2.3 *Concentrated Nitric Acid* — Same as in **A-2.1.2**.

A-5.2.4 *Silver Nitrate Solution* — 10 percent (*m*/*v*).

A-5.2.5 *Standard Chloride Solution* — Dissolve 0·164 g of sodium chloride in water and make up the volume to 1 000 ml. Dilute 100 ml of the solution to 1 000 ml. One millilitre of the solution contains 0·01 mg of chloride (as Cl).

A-5.3 Procedure — Dissolve 0·500 g of the material in 15 ml of dilute nitric acid in a small conical flask. Add 6 ml of hydrogen peroxide and digest on a steam bath until the solution is colourless. Wash down the sides of the flask with water, digest again for 15 minutes, cool and dilute to 250 ml with water. Pipette out 10 ml of the solution into a Nessler cylinder, dilute to about 25 ml with water, add 1 ml of concentrated nitric acid and 0·5 ml of silver nitrate solution. Dilute to 50 ml and mix well. Carry out a control test in another Nessler cylinder using 8 ml of standard chloride solution for pure grade and 4 ml of standard chloride solution for analytical reagent grade and the same quantities of the other reagents.

A-5.3.1 The material shall be taken as not having exceeded the limit given in Table 1 if the turbidity produced with the material is not greater than that produced in the control test.

A-6. TEST FOR BROMATES

A-6.1 Reagents

A-6.1.1 *Potassium Iodide Solution* — 10 percent.

A-6.1.2 *Starch Solution*

A-6.1.3 *Dilute Sulphuric Acid* — 1 : 15.

A-6.2 Procedure — Dissolve 1·0 g of the material in 10 ml of water. Add 2 drops of potassium iodide solution, 1 ml of starch solution and 5 drops of dilute sulphuric acid. Mix thoroughly and allow to stand for 10 minutes.

A-6.2.1 The material shall be taken as not having exceeded the limit prescribed in Table 1 if no blue colour is produced.

A-7. TEST FOR IODIDES

A-7.1 Reagents

A-7.1.1 *Carbon Tetrachloride or Chloroform*

A-7.1.2 *Ferric Chloride Solution* — 10 percent.

A-7.1.3 *Dilute Sulphuric Acid* — 1 : 9.

A-7.2 Procedure — Dissolve 1·0 g of the material in 20 ml of water. Add 1 ml of chloroform or carbon tetrachloride, 3 drops of ferric chloride solution and 5 drops of dilute sulphuric acid. Shake the mixture vigorously.

A-7.2.1 The material shall be taken to have passed the test if no violet colour is produced in the choloroform or carbon tetrachloride layer.

A-8. TEST FOR HEAVY METALS

A-8.1 Apparatus

A-8.1.1 *Nessler Cylinders* — 50 ml capacity.

A-8.2 Reagents

A-8.2.1 *p-Nitrophenol Indicator Solution* — Dissolve 0·2 g of *p*-nitrophenol in hot water and dilute to 100 ml.

A-8.2.2 *Dilute Ammonium Hydroxide* — 1 : 9.

A-8.2.3 *Dilute Acetic Acid* — 1 : 99.

A-8.2.4 *Hydrogen Sulphide Solution* — Saturated and freshly prepared.

A-8.2.5 *Standard Lead Solution* — Dissolve 1·60 g of lead nitrate in water and make up the solution to 1 000 ml with water. Pipette out 10 ml of the solution and dilute again to 1 000 ml with water. One millilitre of this solution contains 0·01 mg of lead (as Pb).

A-8.3 Procedure — Dissolve 2·00 g of the material in 25 ml of water in a Nessler cylinder and add one drop of *p*-nitrophenol indicator solution. Add dropwise dilute ammonium hydroxide until the solution turns yellow. Add dilute acetic acid dropwise until the solution becomes colourless and then add 0·5 ml of the acid in excess. Add 5 ml of hydrogen sulphide solution, dilute to the mark and mix well. Carry out a control test in a similar manner in another Nessler cylinder using 2 ml of standard lead solution for pure grade and 1 ml for analytical reagent grade.

A-8.3.1 The relevant limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

A-9. TEST FOR IRON

A-9.0 Two methods are prescribed for determining iron, namely, Method A and Method B. In case of dispute, Method B shall be the referee method.

A-9.1 Method A

A-9.1.0 *Outline of the Method* — Iron is determined colorimetrically using *o*-phenanthroline. This method requires strict control on the *p*H of the solution.

A-9.1.1 *Apparatus*

A-9.1.1.1 *Nessler cylinders* — 50 ml capacity.

A-9.1.2 *Reagents*

A-9.1.2.1 *p-Nitrophenol indicator solution* — Same as in **A-8.2.1.**

A-9.1.2.2 *Dilute ammonium hydroxide* — 1 : 9.

A-9.1.2.3 *Dilute hydrochloric acid* — 1 : 99.

A-9.1.2.4 *Acetate buffer solution* — Add 23 g of anhydrous sodium acetate to 58 ml of acetic acid (2 M) and dilute to one litre with water. Adjust the final *p*H of the solution to 5·0 ± 0·1 with acetic acid or sodium hydroxide solution (10 percent).

A-9.1.2.5 *o-Phenanthroline reagent* — Thoroughly mix equal volumes of *o*-phenanthroline solution (0·1 percent aqueous), hydroxylamine hydrochloride solution (10 percent aqueous) and acetate buffer solution.

A-9.1.2.6 *Standard iron solution* — Dissolve 0·70 g of ammonium ferrous sulphate in about 100 ml of water containing 10 ml of dilute sulphuric acid (4 N) and dilute to 1 000 ml. Further dilute 100 ml of the solution to 1 000 ml. One millilitre of the diluted solution contains 0·01 mg of iron (as Fe).

A-9.1.3 *Procedure* — Dissolve 1·00 g of the material in 25 ml of water in a Nessler cylinder and add one drop of *p*-nitrophenol indicator. Add dropwise dilute ammonium hydroxide until the solution turns yellow. Add dilute hydrochloric acid dropwise, until the solution becomes colourless and then add 2 ml in excess. Add 5 ml of *o*-phenanthroline reagent, mix well and let it stand for 10 minutes. Dilute to 50 ml and mix well. Simultaneously carry out a control test using 8 ml of standard iron solution for pure grade and 0·2 ml of standard iron solution for analytical reagent grade.

A-9.1.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

A-9.2 Method B

A-9.2.0 *Outline of the Method* — Iron is determined colorimetrically using thioglycollic acid in an alkaline medium. Unlike method A, strict control on *p*H is not required in this case and the method is equally sensitive.

A-9.2.1 *Reagents*

A-9.2.1.1 *Standard iron solution* — Prepare in the same manner as under **A-9.1.2.6.**

A-9.2.1.2 *Thioglycollic acid*

A-9.2.1.3 *Concentrated ammonia* — relative density 0·90.

A-9.2.1.4 *Citric acid solution* — 20 percent (*m*/*v*).

A-9.2.2 *Procedure* — Dissolve 100 g of the material in 25 ml of water in a Nessler cylinder. Add 10 ml of citric acid solution and five drops of thioglycollic acid, mix and make alkaline with ammonium hydroxide. Dilute with water to 50 ml mark and allow to stand for 5 minutes. Carry out a control test using 8 ml of standard iron solution for pure grade and 0·2 ml of standard iron solution for analytical grade.

A-9.2.2.1 The limit prescribed in Table 1 shall be taken as not having been exceeded, if the intensity of colour produced with the material is not greater than that produced in the control test.

A-10. TEST FOR SULPHATES

A-10.1 Apparatus

A-10.1.1 *Nessler Cylinders* — 50 ml capacity.

A-10.2 Reagents

A-10.2.1 *Standard Sulphate Solution* — Dissolve 0·148 g of anhydrous sodium sulphate in water and dilute to one litre in a volumetric flask. One millilitre of the solution contains 0·1 mg of sulphate (as SO_4).

A-10.2.2 *Dilute Hydrochloric Acid* — 1 : 99.

A-10.2.3 *Barium Chloride Solution* — 10 percent.

A-10.3 Procedure — Dissolve 1·000 g of the material in 25 ml of water and transfer to a Nessler cylinder. In another Nessler cylinder carry out a control test using 3 ml of standard sulphate solution for pure grade and 1 ml of standard sulphate solution for analytical reagent grade and 25 ml of water. To each tube add 1 ml of dilute hydrochloric acid and 5 ml of barium chloride solution. Dilute the contents of each tube to the mark and stir well.

A-10.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced with the material is not greater than produced in the control test.

A-11. DETERMINATION OF MOISTURE

A-11.1 Procedure — Weigh accurately about 5 g of the material in a low-form glass-stoppered weighing bottle. Dry at 105 ± 2°C for four hours, cool in a desiccator and weigh.

A-11.2 Calculation

$$\text{Moisture, percent by mass} = 100 \frac{M_1}{M_2}$$

where

M_1 = loss in mass in g on drying, and

M_2 = mass in g of the material taken for the test.

A-12. DETERMINATION OF SULPHATED ASH

A-12.1 Reagent

A-12.1.1 *Concentrated Sulphuric Acid — See* IS : 266-1977*.

A-12.2 Procedure — Weigh accurately about 10 g of the material in a platinum dish. Moisten with 3 drops of concentrated sulphuric acid, ignite gently and moisten again with 3 drops of concentrated sulphuric acid. Reignite to constant mass.

A-12.3 Calculation

$$\text{Sulphated ash, percent by mass} = 100 \frac{M_1}{M_2}$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the material taken for the test.

A-13. TEST FOR ARSENIC

A-13.1 Dissolve 1·00 g of the material in 10 ml of water and carry out the test for arsenic as directed in IS : 2088-1983†, using 0·01 mg of arsenic trioxide for pure grade and 0·001 mg of arsenic trioxide for analytical reagent grade for preparing the comparison stain.

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*Specification for sulphuric acid (*second revision*).

†Methods for determination of arsenic (*second revision*).

A-14. TEST FOR TARRY MATTER

A-14.1 Reagent

A-14.1.1 *Citric Acid*

A-14.2 Procedure — Mix 5·0 g of the material in a dish with 15 ml of water and add 7·0 g of citric acid. Stir until all solids have dissolved.

A-14.2.1 The material shall be taken to have passed the test if no tarry odour is observed.

A-15. TEST FOR BARIUM

A-15.1 Reagents

A-15.1.1 *Dilute Sulphuric Acid* — approximately 4 N.

A-15.2 Procedure — Dissolve 0·5 g of the material in 10 ml of water and add 1 ml of dilute sulphuric acid.

A-15.2.1 The material shall be taken to have passed the test if no turbidity is produced within 5 minutes.

APPENDIX B

(*Clause* 5.1)

PREPARATION OF TEST SAMPLES OF AMMONIUM BROMIDE, PURE AND ANALYTICAL REAGENT

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 The sampling instrument and the containers for samples shall be such that the material has no action on them. They shall be clean and dry.

B-1.2 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature and, as far as possible, the material shall also be protected from light.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In a single consignment of one grade of the material, all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot. If a consignment of one grade of the material is known to consist of different batches of manufacture or of different sizes of containers, then the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

B-2.2 For ascertaining the conformity of the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose (n) shall depend on the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

LOT SIZE N (1)	NO. OF CONTAINERS TO BE SELECTED n (2)
Up to 15	3
16 „ 40	4
41 „ 65	5
66 „ 110	7
111 and above	10

NOTE — In the case of very small lots where the selection of 3 containers may be uneconomical, the method of judging the conformity of the lot to the requirements of the specification shall be agreed to between the purchaser and the supplier.

B-2.3 The containers shall be selected at random from the lot and in order to ensure randomness of selection,random number tables shall be used. In case the tables are not available, the following procedure is recommended for use:

> Arrange all the containers in the lot in a systematic manner and starting from any container, count them as 1, 2, 3........up to r and so on, where r is the integral part of N/n. Every rth container thus counted shall be withdrawn to give sample for tests.

B-3. PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected according to **B-2.3**, a small representative portion of the material of about 100 g shall be drawn with the help of a suitable sampling instrument.

B-3.2 Out of these portions, equal quantities of the material (not more than 90 g) shall be taken and mixed thoroughly to form a composite sample of about 250 g. The composite samples shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.3 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the *n* containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.4 All the individual samples and the composite sample shall be transferred to separate sample containers. All the containers shall be sealed and labelled with full identification particulars.

B-3.5 The referee test samples consisting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of any dispute between the two.

(*Continued from page* 2)

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Printed at Britannia Calendar Mfg Co. Calcutta, India